SYNTHETIC DIOPSIDE

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1. INTRODUCTION

The minerals from the pyroxene group have common formula $\mathbf{XYZ_20_6}$ where **X** is Ca, Mg, Fe^{2+} Mn^{2+}, Na, Li; **Y** - Mg, Fe^{2+}, Mn^{2+}, Fe^{3+}, Al, Cr^{3+}, Ti^{4+} and Z is Si, partly Al and rarely Fe^{3+} [1]. From the pyroxenes the diopside $CaMgSi_20_6$ is the main component of the diopside ceramics and of the white molten stone, while the pyroxene with a diopside structure is the basis for molten basalt and for the basalt glass ceramics [2].

The diopside like the wollastonite improves the quality of the ceramic wares as it increases their bending strength [3-4]. Besides the diopside provides a lower sintering temperature of the masses and therefore it is preferred to the wollastonite.

2. EXPERIMENT AND DISCUSSION

Through crystallization of molten masses or through sintering of mixtures containing (in mass.%): 35-45 quartz sand or silicite and 55-65 dolomite (for diopside) and 45-60 SiO_2 containing rocks (perlite, trass, zeolite rock, diatomite) and 40-55 dolomite (for pyroxene with diopside structure) synthetic pyroxenes are obtained. The quartz sand in the mixtures for diopside can be replaced by silica gel or by silicite when we take into account their ignition loss (IL). The chemical composition of the used raw materials is displayed in table 1.

Rock	Oxides, mass %								Sum, %
	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	IL	
Quartz sand, Senovo	99.75	0.15	0.03	0.04	0.03	-	-	-	100.00
Dolomite, Belovo	1.94	0.16	0.08	19.44	31.70	-	-	46.69	100.01
Perlite, Kardzhali	73.04	13.70	0.86	0.31	0.92	2.77	4.79	3.61	100.00
Trass, Kardzhali	69.52	12.13	0.56	0.37	1.62	1.81	5.06	8.92	99.99
Zeolite rock, Kardzhali	63.72	12.18	0.84	0.68	1.60	0.58	6.64	13.76	100.00
Diatomite, Gen. Toshevo	83.63	5.56	2.13	0.80	0.85	0.59	0.84	5.60	100.00
Silicite, Opaka	96.08	0.60	0.43	0.20	0.59	0.39	0.42	1.30	100.01

Table 1. Chemical composition of the initial rocks

For the synthesis of diopside and of pyroxenes with diopside structure through crystallization of melts, the ground and homogeneous mixtures are put in corundum crucibles and are melted in air environment at 1450°C - 1h. The crystallization of the melts is carried out through their chilling with a hold at 950°C for 1h. Under these conditions the melts from mixtures containing perlite, trass, zeolite rock or diatomite became as hard as glass. Mass crystallization was observed in the melts that contain quartz sand and dolomite as well as in those that contain silicite or silica gel.

For the synthesis of diopside and of pyroxenes with diopside structure through sintering, the ground and homogeneous mixtures are granulated through a sieve 0.5 mm and are thermally treated in the range 1200-l375°C. Parallel to this dry pressed tablets have been fired from the mixtures. As a result in the liquid phase sintering, all the examined masses turned out to be mass crystallized. Maximum production of diopside (≈ 100%) is obtained by mixtures that contain (in mass. %): 38 quartz sand and 62 dolomite; 40 silicite and 60 dolomite at sintering temperature 1325-l350°C for 15-30 min. By increasing the quantity of the quartz sand or the silicite glass appears; while increasing the quantity of the dolomite leads to appearance of melilite presented by akermanite $Ca_2MgSi_2O_7$: 0.287-0.176-0.309 nm (4-0681).

Maximum production of pyroxene with diopside structure (75-95%) is obtained from mixtures containing (in mass %): 55 perlite, trass or zeolite rock, and 45 dolomite; 45 diatomite and 55 dolomite at sintering temperature 1225-1275°C and a hold at that level for 15-30 min. Glass, quartz and melilite are observed as second phases.

The wide isomorphism in the pyroxenes allows us to prognosticate their composition and the presence of impure minerals through P. Niggli's normative – molecular method. The composition of the pyroxenes under the form of normative minerals (hypothetical molecules) is displayed in table 2.

Nº	Mixture, (mass. %) *	Normative minerals, %					Sum, %
		$NaAlSi_2O_6$	$CaAl_2SiO_6$	$CaFe_2SiO_6$	$Mg_2Si_2O_6$	$Ca_2Si_2O_6$	
1	38 quartz sand & 62 dolomite	-	0.4	0.2	46.0	53.4	100.0
2	55 perlite & 45 dolomite	10.0	7.9	1.1	39.0	42.0	100.0
3	45 diatomite & 55 dolomite	2.7	5.5	1.4	43.4	47.0	100.0
4	40 silicite & 60 dolomite	1.8	-	0.4	45.0	52.8	100.0

** Production of pyroxene (quantity of the diopside component $CaMgSi_2O_6$ in the pyroxene), in mass.%: 1 -100 (99.4); 2-75 (81); 3-95 (90.4); 4-100 (97.8).*

Table 2. Composition of the pyroxenes

The X-ray diffractograms of the diopside (1 and 4) and of the pyroxenes with diopside structure (2 and 3) are displayed in table 3.

Synthetic diopside (1 and 4)		Synthetic pyroxene with diopside structure (2 and 3)		Diopside (9-460)	
d, nm	I/I_1, %	*d*, nm	I/I_1, %	*d*, nm	I/I_1, %
0.443	10	0.440	10	0.44	5
0.332	10	0.334	20	0.33	5
0.322	80	0.322	32	0.323	80
0.298	100	0.308*	22*	0.298	100
0.294	75	0.297	100	0.294	70
0.288	45	0.295	80	0.289	10
0.256	35	0.288	40	0.256	10
0.253	35	0.286*	31*	0.253	40
0.229	15	0.255	20	0.229	10
0.2143	25	0.252	30	0.2146	20
0.2123	25	0.248*	25*	0.2124	20
0.2097	10	0.2291	20	0.2101	30
0.2029	15	0.2144	10	0.2000	30
0.1823	20	0.2123	15	0.1830	5
0.1746	35	0.2100	10	0.1748	40
0.1620	30	0.2040*	20*	0.1622	20
0.1546	5	0.2030	20	0.1548	5

Table 3. Diffractograms of the pyroxenes

In table 3 the lines of the akermanite are marked with *. Connected to the diffractogram (2 and 3) are also 0.1823(12), 0.1759*(18)*, 0.1745(15), 0.1619(12) and 0.1545(5).

3. CONCLUSION

Diopside from quartz sand (silica gel or silicite) and dolomite has been synthesized through crystallization of molten masses and through sintering. The production of diopside is ≈ 100%. Taking into consideration the possibilities for isomorphic substitutions in the pyroxene we have obtained by sintering pyroxenes with diopside structure from SiO_2-containing rocks (perlite, trass, zeolite rocks, diatomite) and dolomite. The production of pyroxene varies from 75 to 95%.

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